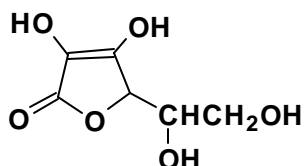


Chemistry 119: Experiment 7

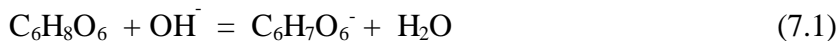
Potentiometric Titration of Ascorbic Acid in Vitamin C Tablets

Vitamin C is another name for ascorbic acid ($C_6H_8O_6$, see below), a weak acid that can be determined by titration with 0.1 M NaOH. Because ascorbic acid is a fairly weak acid ($pK_a = 4.17$), the titration will be successful only when no other acids are present in the sample. Other acids would obscure the end point of the acid-base titration.



Ascorbic acid in tablet form is sold as vitamin C. These tablets generally contain only ascorbic acid and some **excipients** - additives that make the delivery of the active ingredient simpler. Here, the excipients are inert materials that simplify pressing the solid into tablets. We should therefore be able to titrate vitamin C tablets directly, without worry of interference from other acids in the tablet.

We will determine the concentration of ascorbic acid in some samples of brand-name and "generic" vitamin C tablets. The reaction we will use for the titrimetric analysis of vitamin C tablets is the simple acid-base reaction



To detect the equivalence point of this titration, we will use a pH titration.

A pH titration relies on the fact that the voltage developed at a glass electrode depends primarily on the activity of the hydronium ions in solution around the electrode, according to the Nernst equation

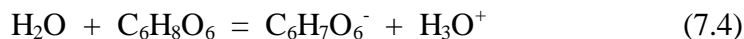
$$E_{glass} = k + \frac{2.303RT}{F} \log(a_{H^+}) \quad (7.2)$$

or

$$E_{glass} = k - \frac{2.303RT}{F} pH \quad (7.3)$$

Since the "pH" meter is measuring the voltage at the glass electrode (in mV), but the meter reads in pH units, the meter must be calibrated in order to determine the unknown quantities k and T . This is done by using solutions of known pH, *i.e.*, standard acid-base buffers.

Once the meter is calibrated, the pH of the ascorbic acid solution is easily followed as a function of the added NaOH. The electrodes are responding only to the H_3O^+ ions involved in the equilibria:



Addition of the OH^- alters the amounts of $C_6H_8O_6$ and $C_6H_7O_6^-$ present which, in turn, affects the hydronium ion concentration, and the pH.

Above pH 10.5 to 11, the glass electrode begins to respond to other ions (mainly to Na^+ ions here), since so few H_3O^+ ions remain and because $[\text{Na}^+] \sim 10^9[\text{H}_3\text{O}^+]$. This effect is called the alkaline error since it *appears* that more hydronium ions are present (due to the response of the electrode to the Na^+ ion) than really are. Thus, there is no reason to carry the titration beyond pH 10.5, as we do not measure anything of interest to us above that point.

Prelaboratory Assignment

A sample of ascorbic acid (FW = 176.13) weighing 0.4964 g was titrated with 0.1025 M NaOH. If the equivalence point occurred after 13.11 mL of base were added, calculate the weight percent of ascorbic acid in the sample.

Apparatus

stirrer and stir bar
pH meter and combination electrode
2 250-mL beakers
50-mL burette
25-mL graduated cylinder
stirring rod
watch glass

Chemicals

standard NaOH (0.1 M, Experiment 4)
pH buffers (4 and 7)
phenolphthalein indicator
ascorbic acid unknown

Procedure:

You should work in PAIRS.

1. Weigh out to the nearest ± 0.1 mg a vitamin C tablet and place it in a clean, dry 250-mL beaker.
2. **Carefully** crush the vitamin C tablet in the beaker with a stirring rod. Vitamin C will oxidize in air, and it is important to use fresh tablet samples in the analysis. Therefore, close the bottle containing the tablets tightly after getting a sample.
3. Dissolve the vitamin C sample in about 100 mL of distilled water. Warm as necessary to effect dissolution of the vitamin C. Recall that the binder particles will not dissolve.
4. Clean and rinse the 50-mL burette, then fill it with the NaOH solution prepared in Experiment 4 above.

5. Calibrate the pH meter according to the instructions provided by the teaching assistant using the pH 4 and pH 7 standard buffers. The electrode assembly should be rinsed with distilled water between measurements. Do not dry the electrode.
6. Place a stirring motor under the electrode, leaving 2 to 3 inches of extra space. Then position the electrode in the 250-mL beaker containing the vitamin C solution, and place the beaker on the stirrer.
7. Carefully add the stir bar to the beaker, and arrange the electrode so that the stir bar will miss hitting it when the bar turns. It may be necessary to add distilled water to keep the lower 2 cm of the electrode assembly covered with solution. Carefully start the stirring motor.
8. Make the initial burette reading, then position the burette over the beaker, bringing the burette tip as close as possible to the solution's surface.
9. Add 2 to 3 drops of phenolphthalein indicator and begin the titration. Add small increments of base, reading both the stabilized pH and volume after every addition. Add enough base to cause changes in pH of about 0.2 pH units. Try to make readings at roughly equal pH increments.
10. When the pH changes by more than 0.4 unit or when you come within 1.5 mL of the pre-calculated equivalence point, reduce the size of the NaOH additions. As you near the equivalence point, the pH will change considerably upon the slightest addition of base. You may want to use the stirring rod to transfer titrant from the burette to the solution; this will enable you to add quarter drops.
11. As the titration progresses through the equivalence point, note the volume and pH where the indicator changes color (visual end point). It should be near where the change in pH is greatest. The difference between the true **equivalence** point and the endpoint, where the indicator changes color, is called indicator error. When the indicator is chosen correctly, this error will be small.
12. Once past the equivalence point, the pH changes will again be small. Increase additions of NaOH to save time, but keep pH changes below 0.1 pH units. Stop the titration at pH 10.5-11.
13. Rinse the electrode, then repeat the analysis for a new vitamin C tablet. (Do not lose the stir bar in the sink drain when disposing of the titrated solution).
14. Rinse the electrode.
15. Return the electrode to the beaker containing pH 4 buffer. Do not let the electrode rest on the bottom of the beaker.

Calculations

1. Make a graph of pH versus the volume of base added for the titration of ascorbic acid with NaOH. Graph appearance will be graded!
2. Estimate the volume of the equivalence point from the graph of the NaOH-ascorbic acid titration. With care, you should be able to obtain 2 significant figures beyond the decimal.
3. For the titration make a table of changes in pH and in volume. Take successive pairs of readings: (pH₁ and pH₂) and (V₁ and V₂). Include columns giving the change in pH per change in volume ($\Delta\text{pH}/\Delta V = (\text{pH}_2 - \text{pH}_1)/(\text{V}_2 - \text{V}_1)$) and the average volume ($\bar{V} = (\text{V}_1 + \text{V}_2)/2$). An example table is shown in Table 7.1.
4. From entries in your table, plot $\Delta\text{pH}/\Delta V$ versus \bar{V} (the last two columns) for the titration. This generates a first-derivative plot for the titration curve. The equivalence point should now appear as a peak on this plot. The appearance of this graph will also be graded.
5. From the peak in the first-derivative plot, estimate the volume for the equivalence point. By comparing the volume obtained here with that observed from the pH vs. volume plot, ensure that your volume estimate is reasonable. Again, attempt to obtain two significant figures beyond the decimal point.
6. Use the equivalence point volume obtained from the first-derivative plot to calculate the millimoles of the ascorbic acid in the dissolved tablet sample.
7. Now use the end point volume from the indicator change to calculate the millimoles of ascorbic acid in the tablet sample.
8. From the formula weight of ascorbic acid, determine the weight of ascorbic acid in the tablet sample using the results from steps 6 and 7 above.
9. Determine the weight percent of ascorbic acid in the tablet from the weights obtained in step 8 above.

Table 7.1 Example Data from the Titration of a Weak Acid with 0.100 M NaOH

| Vol. NaOH Added (mL) | pH | $V_2 - V_1$ | $\text{pH}_2 - \text{pH}_1$ | $\frac{\text{pH}_2 - \text{pH}_1}{V_2 - V_1}$ | $\frac{V_1 + V_2}{2}$ |
|---------------------------------|-----------|-------------------------------|---|---|---|
| 38.20±0.02 | 5.77±0.02 | | | | |
| | | 0.11±0.03 | 0.13±0.03 | 1.2±0.4 | 38.255±0.014 |
| 38.31 | 5.90 | 0.09 | 0.19 | 2.1±0.8 | 38.335 |
| 38.40 | 6.09 | 0.08 | 0.31 | 3.9±1.5 | 38.440 |
| 38.48 | 6.40 | 0.12 | 1.49 | 12.4±3.1 | 38.540 |
| 38.60 | 7.89 | 0.09 | 1.91 | 21.2±7.0 | 38.645 |
| 38.69 | 9.80 | 0.12 | 0.38 | 3.2±0.8 | 38.750 |
| 38.81 | 10.18 | 0.09 | 0.12 | 1.3±0.5 | 38.855 |
| 38.90 | 10.30 | | | | |

10. The pH at the half-titrated point (where the volume of titrant added is half the end point volume) should be equal to the pK_a of the acid. Compare your result with the expected value.
11. Using the report form, turn in your two graphs, your molarity of NaOH, and your values for the weight of ascorbic acid in the tablet and the weight percent of ascorbic acid in the tablet.

This experiment has been adapted from a laboratory manual authored by Professor S. D. Brown.

Version 11.97

Chem 119
Report Sheet: Experiment 7
Analysis of Vitamin C Tablets

Name: _____

TA: _____ Section: _____

NaOH Molarity = _____ M

pH Titration

1 2

wt tablet (g) _____

vol. NaOH _____ (from first-derivative of pH vs. volume NaOH)

wt $C_6H_8O_6$ (g) _____

% $C_6H_8O_6$ (w/w) _____

Avg. % $C_6H_8O_6$ (w/w) _____

Std. dev. % $C_6H_8O_6$ (w/w) _____

RSD _____ %

Phenolphthalein Endpoint

1 2

wt tablet (g) _____

vol. NaOH _____ (for phenolphthalein endpoint)

wt $C_6H_8C_6$ (g) _____

% $C_6H_8C_6$ (w/w) _____

Avg. % $C_6H_8C_6$ (w/w) _____

Std. dev. % $C_6H_8C_6$ (w/w) _____

RSD _____ %

(attach graphs as indicated above)